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Influence of voids on the flexural resistance of the NCF/RTM6 composites

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Abstract

A major difficulty to achieve maximum weight savings in the manufacture of composite structural components, is the tendency of these materials have the formation of voids and cracks in the interior and surface components. In aeronautical applications, controlling the volume fraction of fibers, resins and empty the components of composite is very hard. In this work, composites of epoxy matrix RTM6 reinforced with NCF (non crimp fabric carbon) processed by resin transfer molding (RTM) were characterized for porosity (P_{ap}) and density (ρ_{ap}). We used a method based on Archimedes' principle (ASTM C830) and the technique of helium pycnometer. The porosity values were compared with those determined by acid digestion (ASTM D3171). The mechanical properties of processed composites was evaluated by testing on the performing flexural and the results were correlated with the porosity value. All techniques tested to determine void content are satisfactory. The differents results can be justified for heterogeneous void distribution on laminate and differences among techniques characteristics.

Keywords: Composites; Flexural test; RTM; Voids.

1. Introduction

Currently the technological interest in the use of the composites is increasing, due to the requirement of lighter higher quality materials, essential factors to fulfill the specifications of projects and to reduce

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the overall operational costs. The technology of fibrous composites is very important for the accomplishment of projects in which the parameter weight must be considered, especially in the aeronautical industry. With the production of high performance structural composites, following the criteria such as security, reproducibility and low cost, the national industry has shown an increasing interest for the process of molding using resin transfer (RTM). RTM is a manufacturing process of in the resin is injected or drawn into a mold, which contains the fibres, from a homogeniser under low pressure.

The mold can be made from composites for low production cycles or with aluminium or steel for larger production. It is a promising process for structures of polymers composites with a complex geometry, that demand high quality finishing and no size limitation, presenting a consistent thickness. Considered the inherent disturbances to the tool rack and the process, it is possible to assure success, mainly in respect to the minimization of the formation of voids in the part produced. The formation and the growth of voids cause a reduction in resistance, rigidity and useful life due to fatigue of the laminate, resulting in catastrophic failure of the part. It is known that the resistance of the composites to some types of stress (interlaminar shear and compression, among others) is reduced with the increase of the volume of voids [1-3]. A significant fact is that voids favor water absorption of the polymer matrix, implying in a potential increase of the existing voids [2,4,5]. The formation of voids in the interior of organic matrix composites is associated to two causes: incomplete impregnation of the matrix fiber, resulting in air retention in the interior of the filaments and volatile substance release present in the components of the the matrix, occuring during the polymerization [6]. The voids can be elements of cracks in the microstructure of the composites, so attention must be given in relation to its quantity (indicated by the volume of voids) and to its form. Voids of spherical form almost always are in the interior of the layers, while voids of prolonged form are located in the interface in between the layers of the composite, causing interlaminar defects [7]. Therefore, the control of voids in aerospace structures is very important [8]. The mechanical properties are influenced by quantity, shape and location of voids, that can be different from sample to sample [9,10]. This difference can influence whether or not a crack emanates from a void [10]. The void content has an important effect on the mechanical properties of composites such as interlaminar shear strength, compressive strength, modulus and bending properties [10,11]. Thus, composite parts, especially structural parts, must be inspected after manufacturing to detect and quantify defects, allowing a safe use [11]. Some detection methods as apparent density, optical microscopy and acid digestion have been used for industries and researchers, being preferred non-destructive methods [8]. The apparent density and acid digestion techniques are used to quantify voids. The knowledge of void is an indication of the quality of composites [12].

2. Materials and Methods

2.1. Composites

Cured carbon fiber multiaxial non-crimp reinforcement/RTM6 composite panels supplied by Hexcel Composites Co. were used. The laminate panels were prepared by RTM processs in 5 different batches. For this, seven cross ply multiaxial $[45/0/\text{-}45/90]_{2s}$, 60% fiber volume faction nominal, were processed. Each batch, designated as IRXXX, produced one composite panel that provided the specimens.This reinforcement is a combination of Hexcel intermediate modulus IM7-12k carbon fiber non-crimp quadri-axial orientated at $[+45/0/\text{-}45/90]_{2S}$, with a real weight of 772 g/m², stitched by a polyester yarn.

2.2. Porosity and apparent density (Archimedes' principle)

In the calculation of the apparent specific mass, based on the principle of Archimedes (ASTM C830) [13], we determined the masses of the samples under the following conditions: Dry mass ($\boldsymbol{m_s}$), after drying the material in greenhouse under 110ºC for a period of 2 h; Immersed mass ($\boldsymbol{m_i}$), after immersion of the samples in distilled water, boiled during 2 h and kept at rest for a minimum period of 12 h; Humid mass (***m_u***), after the excess water contained in the sample was removed. All the measurements had been taken at room temperature with using an analytical scale. With the use of this method we also determined the apparent porosity, by means of the expressions (1) and (2):

$$P_{ap} = \frac{m_u - m_s}{m_u - m_i} \times 100 \quad (1) \qquad \rho_{ap} = \frac{m_s}{m_u - m_i} \quad (2)$$

2.3. Helium pycnometer

We carried out the pycnometry analysis of helium for the determination of the real density of the composites. The analysis had been carried using Micromeritics, model Multivolume 1305, leased by the Associated Laboratory of Combustion and Propulsion of the National Institute of Pesquisas Espaciais (LCP-INPE) of Cachoeira Paulista/SP.

2.4. Matrix digestion using sulfuric acid/hydrogen peroxide

The NC2/RTM6 Hexcel composites was previously tested under flexural solicitation for this research group. Non delaminated parts of one specimen test was choose to acid digestion analyze made in a Marconi Digestor. Following ASTM D 3171 (procedure B), was used 60 mL of sulfuric acid and 30 mL of H_2O_2 50%. The temperature was maintained at 150°C for 3 hours. The residual fibers were washed with distillate water and acetone. After dried for 1.5 hour at 100°C, the fibers was weighed again to determinate de final weigh and allow a comparison with initial composite weigh. This data were determinate using equations available in ASTM D3171 described in equations (3), (4) and (5) [12].

$$V_r = \left(\frac{m_f}{m_i}\right) \times \left(\frac{\rho_c}{\rho_r}\right) \times 100 \quad (3) \qquad V_m = \left(\frac{m_i - m_f}{m_i}\right) \times \left(\frac{\rho_c}{\rho_m}\right) \times 100 \quad (4) \qquad V_v = 100 - \left(V_r + V_m\right) \quad (5)$$

Where: V_r = reinforced fiber volume; Vv = void volume; V_m = matrix volume; m_f = mass final; m_i = mass initial; ρ_c = composite density; ρ_m = matrix density; ρ_r = reinforced fiber density

2.5. Mechanical Tests

The bending tests were performed in an universal test machine INSTRON 880 with the loading nose in one point, according to specification ASTM-D790. The flexural modulus was calculated from 20 specimens obtained from the five different composite panels. The dimension of the specimen was 48 mm length, in that way to comply with the 16:1 ratio between the length and thickness of the specimen [14].

3. Results

3.1 Porosity and apparent density (Archimedes principle)

The results of the apparent porosity and density present in the samples of the composite analyzed using the Archimedes' Principle are shown on Table 1. Three samples of the set IR 384, IR 385, IR 386, IR 387, IR 388, IR 389 and IR 390 were used and the measurements were checked in triple. The average of the apparent porosity found was of 0.50%. It is to be noted that this technique is only able to detect the superficial porosity. Thus the low amount of the composite porosity.

Table 1. Results of apparent porosity (P_{ap}) and apparent density (ρ_{ap}) found using a technique based on the Arquimedes principle (ASTM C830).

Samples	IR 384	IR 385	IR 386	IR 387	IR 388	IR 389	IR 390
P_{ap} (%)	0.46	0.50	0.51	0.53	0.52	0.51	0.48
ρ_{ap} (g/cm^3)	1.548	1.491	1.538	1.532	1.529	1.536	1.499

3.2. Helium pycnometer

The results of the real density of the composite analyzed using the Helium Picnometry technique is shown on Table 2. The equipment has carried out 10 reading runs, where possible, and based on the average obtained through these readings, we determined the real density (Max and Min density) of the composite sample. As it can be observed, the value of the real density was very close the apparent density determined through the method based on Archimedes' principle.

Table 2. Results of real density determined using Helium Pycnometer.

Samples	IR 384	IR 385	IR 386	IR 387	IR 388	IR 389	IR 390
Real Density (g/cm^3)	1.586	1.570	1.618	1.611	1.584	1.619	1.567
Maximum Density (g/cm^3)	1.590	1.574	1.626	1.613	1.588	1.629	1.570
Minimum Density (g/cm^3)	1.581	1.566	1.611	1.609	1.580	1.609	1.564

3.3. Matrix digestion

Table 3 lists the measurement of acid digestion, fiber and void percentual volume. This data were determinate using equations available in ASTM D3171 described in equations (3), (4) and (5) [12]. For all calculations were considered ρ_c = 1.524 g/cm^3 (theoretical value), ρ_m = 1.14 g/cm^3 and ρ_r = 1.78 g/cm^3.

Table 3. Measurement of void and fiber contents for NC2/RTM6 composite.

Samples	IR 384	IR 385	IR 386	IR 387	IR 388	IR 389	IR 390
Fiber Volume (%)	63.62	63.73	63.38	63.43	72.17	73.37	62.64
Void Volume (%)	2.04	2.09	1.89	1,93	1.97	2.02	1.48

3.5. Mechanical Tests

Table 4 shows the variation of the flexural modulus for the 20 specimens analised. The maximum variation of the tests in a laminate family (batch) was 10.9%, attributed to the low values determined in two specimens in batch IR389. However, the other families present low dispersion of the results, with a minimum variation of 4.9%. The variations observed are narrow considering the laminate stacking sequence and the elevate number of interfaces, in which more defects are introduced during the manufacture. Moreover, in respect of the average values, the results are very close and the standard deviations varied less than 3.03 GPa, which value was a consequence of one low value (24.41 GPa) in batch IR 389. Other values did not show significant differences as verified in their standard desviaton values.

Table 4. Flexural modulus

	Results (GPa)						
Batch / Specimens	**IR384**	**IR385**	**IR386**	**IR387**	**IR388**	**IR389**	**IR390**
1	31.88	32.95	32.93	26.36	32.35	24.41	27.54
2	24.25	30.15	29.89	27.19	31.73	30.03	29.84
3	30.71	27.95	30.96	28.18	27.97	26.44	26.97
4	29.76	28.82	28.55	31.68	31.41	30.86	29.33
Average	**29.15**	**29.97**	**30.58**	**28.35**	**30.86**	**27.93**	**28.42**
Minimum	**24.25**	**27.95**	**28.55**	**26.36**	**27.97**	**24.41**	**26.97**
Maximum	**31.88**	**32.95**	**32.93**	**31.68**	**32.35**	**30.86**	**29.84**
Standard Deviation	**3.37**	**2.18**	**1.85**	**2.34**	**1.97**	**3.03**	**1.38**
Variation	**11.6%**	**7.3%**	**6.0%**	**8.3%**	**6.4%**	**10.9%**	**4.9%**

As observed in Figure 1, the load x displacement curve of the composite specimens tested presents elastic behavior from the begining to the end of the curve and the fracture ocurred at the maximun load. Despite the fact that the tests were conducted in small span-to-depth ratio, the curves in Figures 1 and 2 did not present nolinear zone. This phenomenon is explained by local deformation effects which mean a change in contact area between the specimen and the load cylinder.

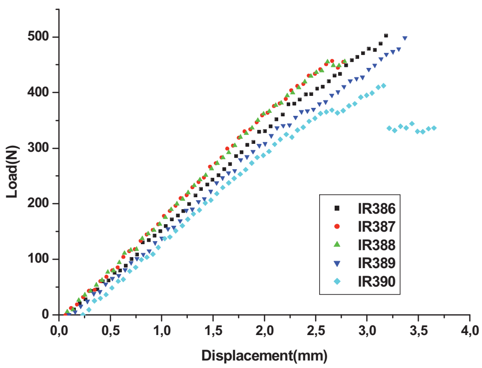

Fig. 1. Load-displacement curves.

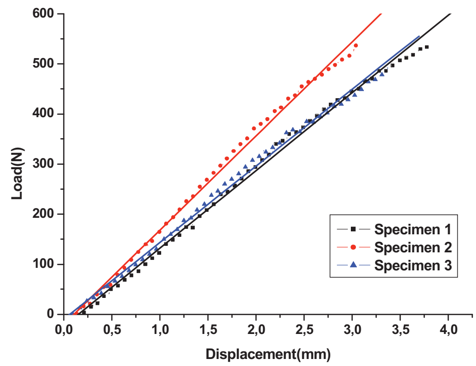

Fig. 2. Load-displacement curve of IR 389 specimens.

The curves of the three specimens IR389 that presented great differences were plotted, according to Figure 3. It is possible to verify that specimen 1 and 3 present lower value than the rest of the specimens contributing to increase the standard deviation.

The fiber volume described in Table 3 for sampling IR 386 can be used to theoretical calculation of Young´s Modulus (E). According to values averages 63.38 % (fiber) and 34.73% (resin) and considering E_f =276 GPa and E_m= 3.3 GPa the result was E= 69.15 GPa, higher that found with flexural test E= 29.4 GPa. Difference between E values can be justified for void presence and their effects, not considered in E theoretical. Other factors as interfacial adhesion can induce decrease in E value because the tension cannot be transfer accordingly.

4. Conclusions

All techniques tested to determine void content are satisfactory. The differents results can be justified for heterogeneous void distribution on laminate and differences among techniques characteristics.

The technique using the Archimedes' Principle provided us with the amount of voids at surface level, thus the low amount detected. The image analysis technique was able to detect all the pores, those that had a connection to the surface and those that did not (bubbles). With regards to determining the density of the composite, all the techniques used were satisfactory hence the similarity of all the results obtained.

During the mechanical tests it was verified good regularity in the rupture behavior, without catastrofic failures of the laminates, thus representing high confiability and reproductibility on the used RTM process. Due to the high resistance to damages, it was possible to reach high values of flexural modulus. The stitch of polyester fiber within the carbon fabrics was identified as one of the weakness points of the preform, which acts as a stress concentration as observed in many tested specimens.

The Young´s Modulus calculated with fiber fraction was superior that value of flexural test. This can be explained because the E theoretical not consider void effects.

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